



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN THE APPLICATION OF

Michael Cole

SERIAL NO: 09/550.693

FILED: April 17, 2000

FOR: Evaporation of Liquids

) Examiner: Virginia Manoharan

)

) Group Art Unit No. 1764

)

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I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail in an envelope addressed to "Commissioner of Patents, Washington, D.C. 20231", on June 5, 2002.

Name of person signing Jennifer J. Ramirez

Signature

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CLAIM FOR PRIORITY

Honorable Commissioner of  
Patents and Trademarks  
Washington, D.C. 20231

Dear Sir:

Under the International Convention, for the purposes of priority, applicant claims the benefit of the following U.K. Applications:

9908747.0, filed April 17, 1999,

9914329.9, filed June 19, 1999,

9918914.4, filed August 12, 1999 and

9930292.9, filed December 23, 1999.

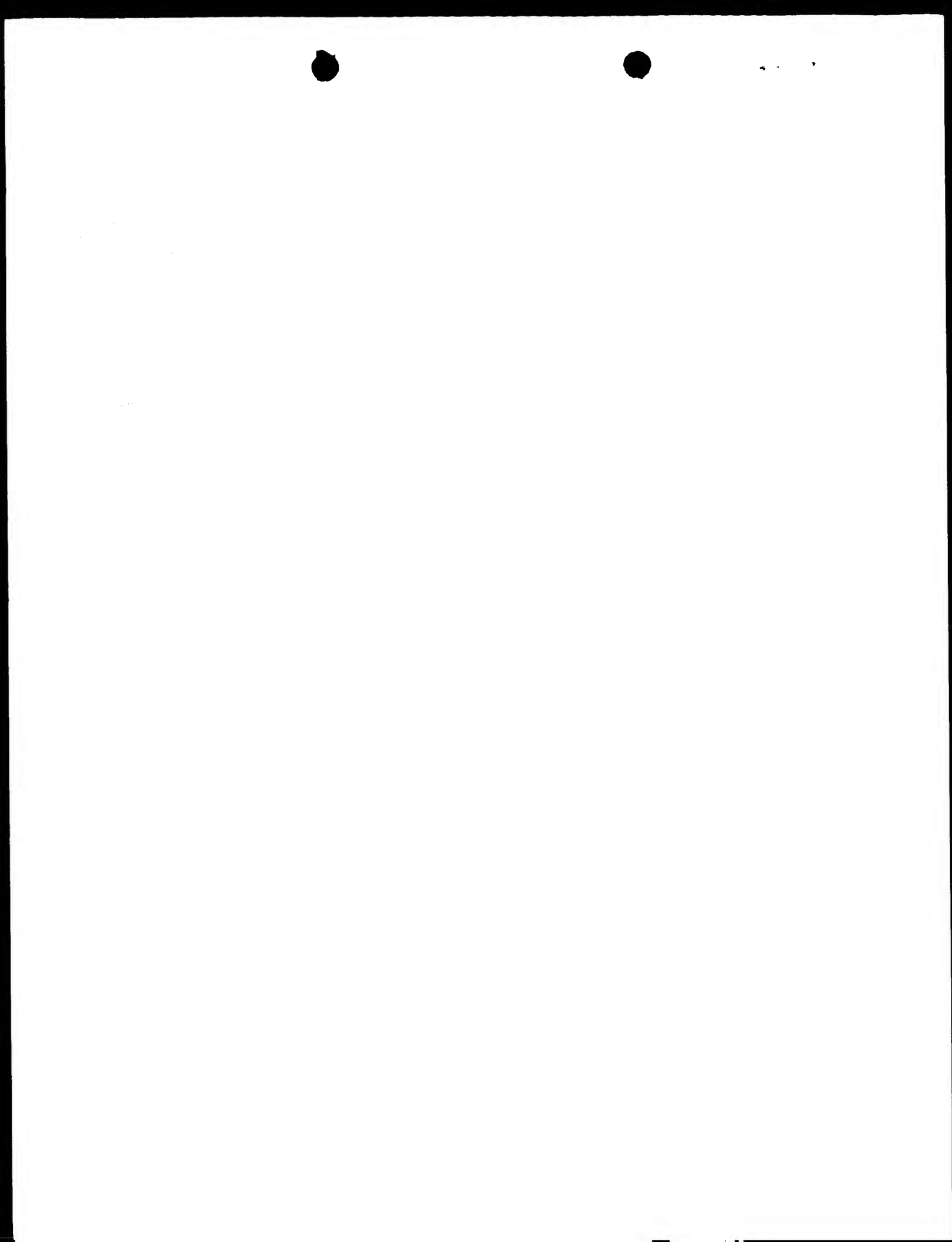
A certified copy of each said application is appended hereto.

DATE: June 5, 2002

Respectfully submitted,

William M. Lee, Jr.  
Registration No. 26,935  
Lee, Mann, Smith, McWilliams  
Sweeney & Ohlson  
P.O. Box 2786  
Chicago, Illinois 60690-2786  
(312) 368-1300  
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The Patent Office  
Concept House  
Cardiff Road  
Newport  
South Wales  
NP10 8QQ

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In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

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1. Your reference

C1067/C

2. Patent application number

*(The Patent Office will fill in this part)***9908747.0**3. Full name, address and postcode of the or of each applicant *(underline all surnames)*Michael COLE  
Poplar Farm  
Low Road  
Marlesford  
Woodbridge  
Suffolk IP13 0ALPatents ADP number *(if you know it)*

If the applicant is a corporate body, give the country/state of its incorporation

07076086002

4. Title of the invention

Evaporation of Liquids

5. Name of your agent *(if you have one)*

Keith W Nash &amp; Co

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*90-92 Regent Street  
Cambridge  
CB2 1DPPatents ADP number *(if you know it)*

1206001 ✓

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and *(if you know it)* the or each application number

Country

Priority application number  
*(if you know it)*Date of filing  
*(day / month / year)*

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application

Date of filing  
*(day / month / year)*8. Is a statement of inventorship and of right to grant of a patent required in support of this request? *(Answer 'Yes' if:*

NO

- a) any applicant named in part 3 is not an inventor, or
  - b) there is an inventor who is not named as an applicant, or
  - c) any named applicant is a corporate body
- See note (d))*

# Patents Form 1/77

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Description

Claim(s)

Abstract

Drawing(s)

0  
2 ✓ 10/1  
0  
0  
2 ✓ 2

10. If you are also filing any of the following, state how many against each item.

Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for preliminary examination and search (Patents Form 9/77)

Request for substantive examination (Patents Form 10/77)

Any other documents (please specify)

11. I/We request the grant of a patent on the basis of this application.

Signature

Date 16.04.99

Keith W Nash & Co, Agents

12. Name and daytime telephone number of person to contact in the United Kingdom

Mr Nash (01223) 355477

## Warning

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

## Notes

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- For details of the fee and ways to pay please contact the Patent Office.

C1067.00/C

Title: Evaporation of Liquids

Field of the Invention

This invention concerns evaporation of liquids.

Background to the Invention

Evaporation of TFA mix or other volatile liquids by directing a stream of air or an inert gas such as nitrogen, onto the surface of a liquid is a well known process, widely used for evaporating volatile liquids. It suffers from the disadvantage that large volumes of inert gas or air, contaminated with vapour, must be disposed of. This can be expensive if the liquid is inflammable or toxic, as is the case with TFA.

Summary of the Invention

According to the present invention, the inverse of this process may be employed, in which gas, rich in vapour from near the surface of a liquid, is continuously removed.

This allows much easier collection of any toxic material.

In the drawings, the known process is shown in Figure 1, and an embodiment of the invention is shown in Figure 2.

In Figure 2 a liquid sample 3 is contained in a tube 2 inside a sealed chamber 1.

A tube 5 is placed with one end 11 in the tube 2 and above the sample 3. This tube passes through the chamber 1 through a sealing means 12, through a further sealing means 13, and finally into a sealed refrigerated condensing vessel 6.

A further tube 8 connects the refrigerated condensing vessel 6 to a pump 7 which draws gas from the vessel 6 and passes it through a further tube 9 back to the chamber 1 again through a sealing means 14.

A pressure control means 15 allows the pressure in the system to be held at any required level by venting gas to atmosphere or admitting gas into the system from a suitable source eg a gas cylinder via a pressure regulator.

In operation the pump reduces the pressure in the refrigerated vessel 6, which causes vapour 4 to be drawn into the vessel 6 together with some gas. The temperature of the vessel 6 is maintained at a low enough value to condense the vapour, and any permanent gas passes through the vessel into the pump 7. Gas extracted by the pump 7 could be discharged to the atmosphere because it has been stripped of undesirable vapours in the refrigerated vessel 6 or, as in this illustration, may be recirculated to continue to evaporate the sample.



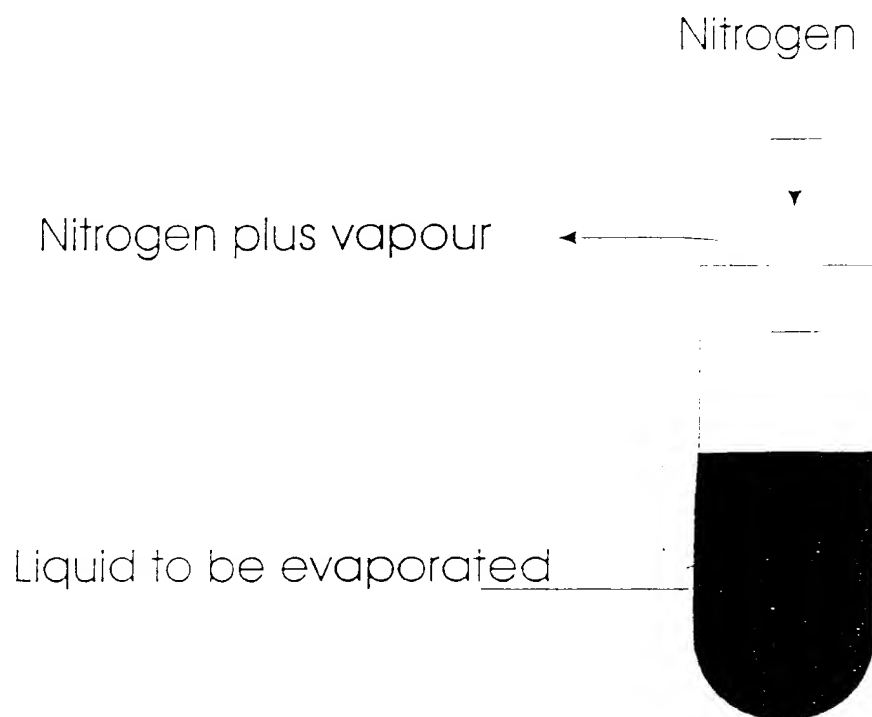


Figure 4- Nitrogen blow-down



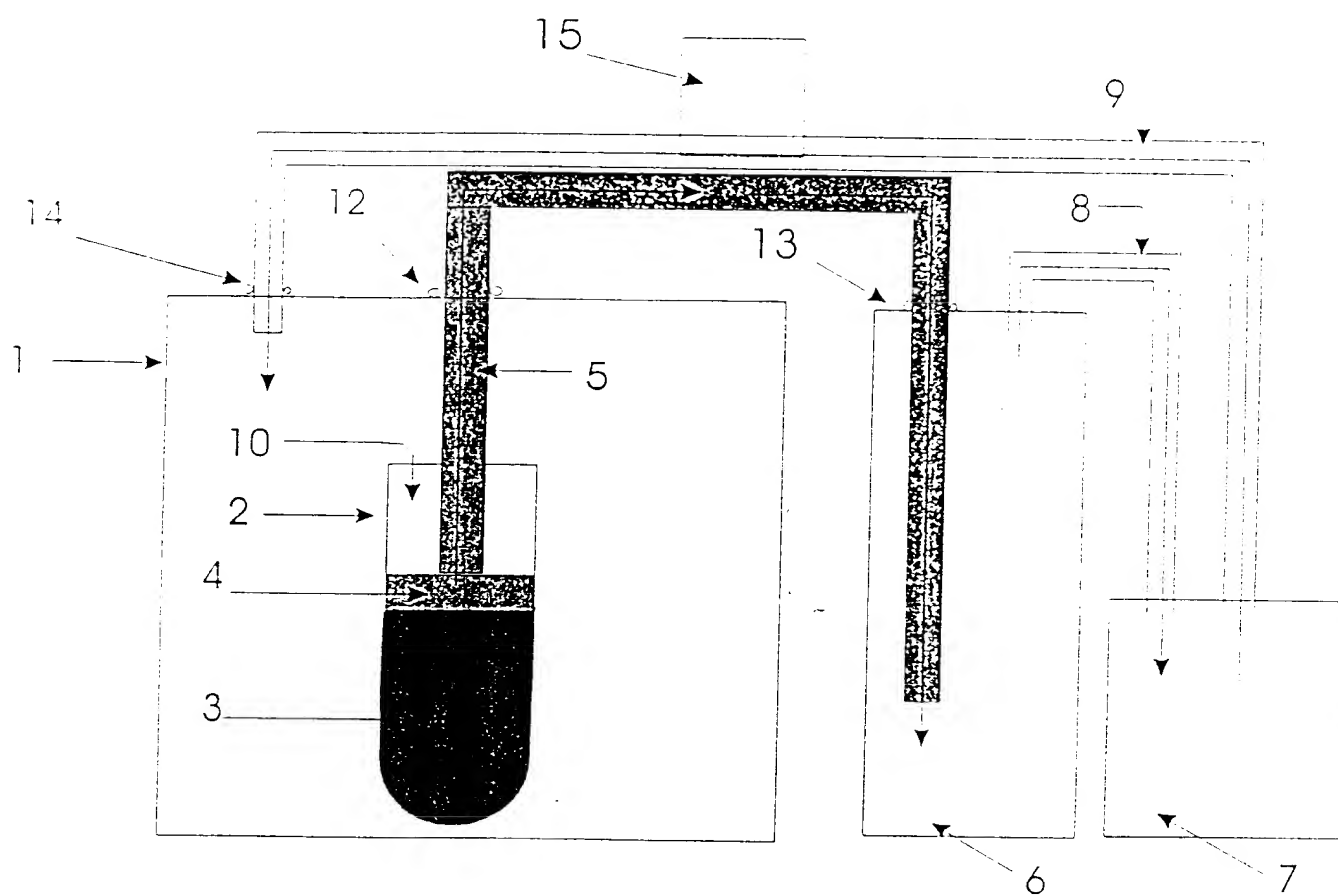
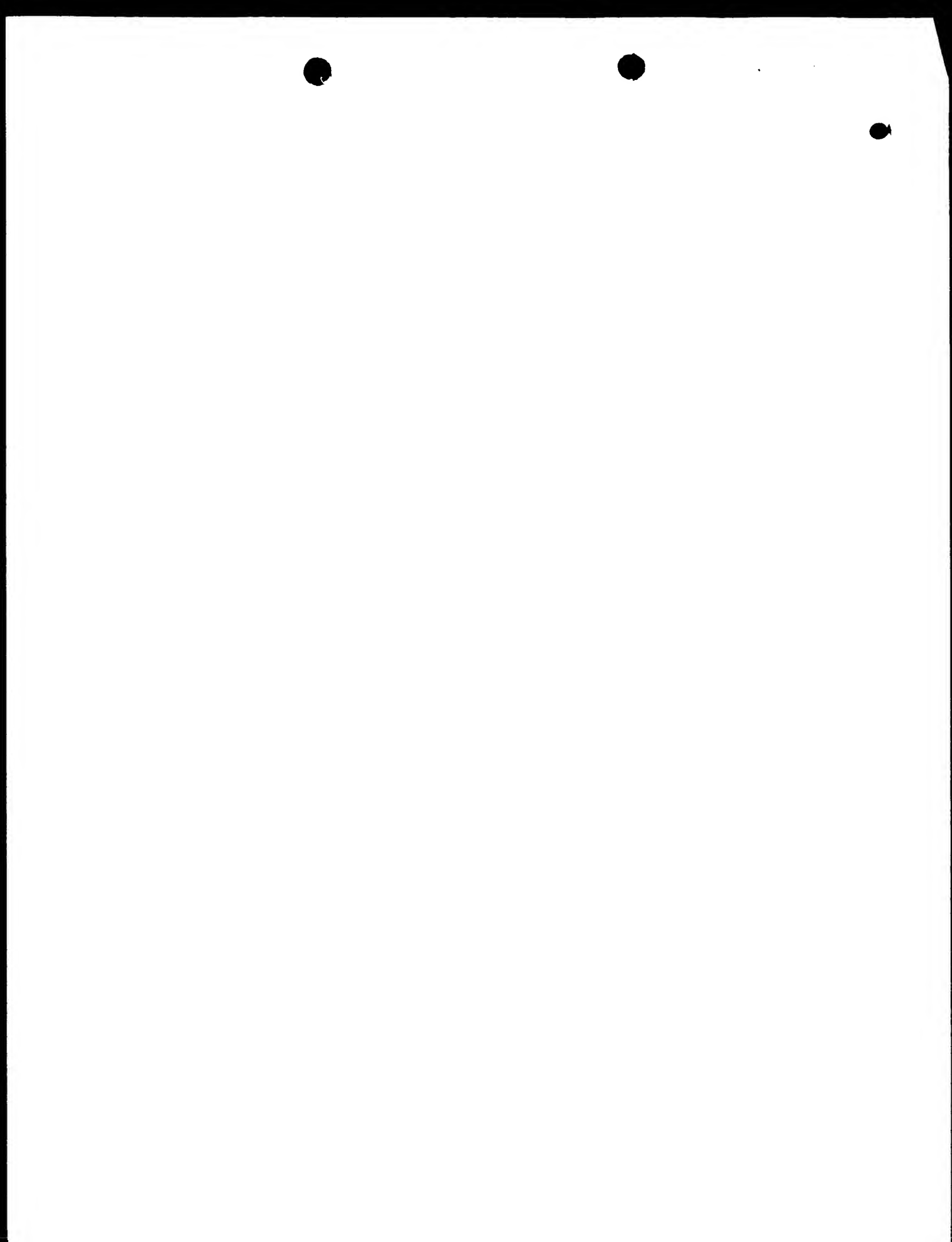


Figure ~~5~~<sub>2</sub> - Evaporation Apparatus





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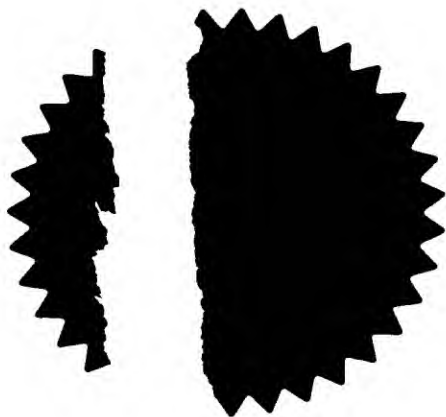
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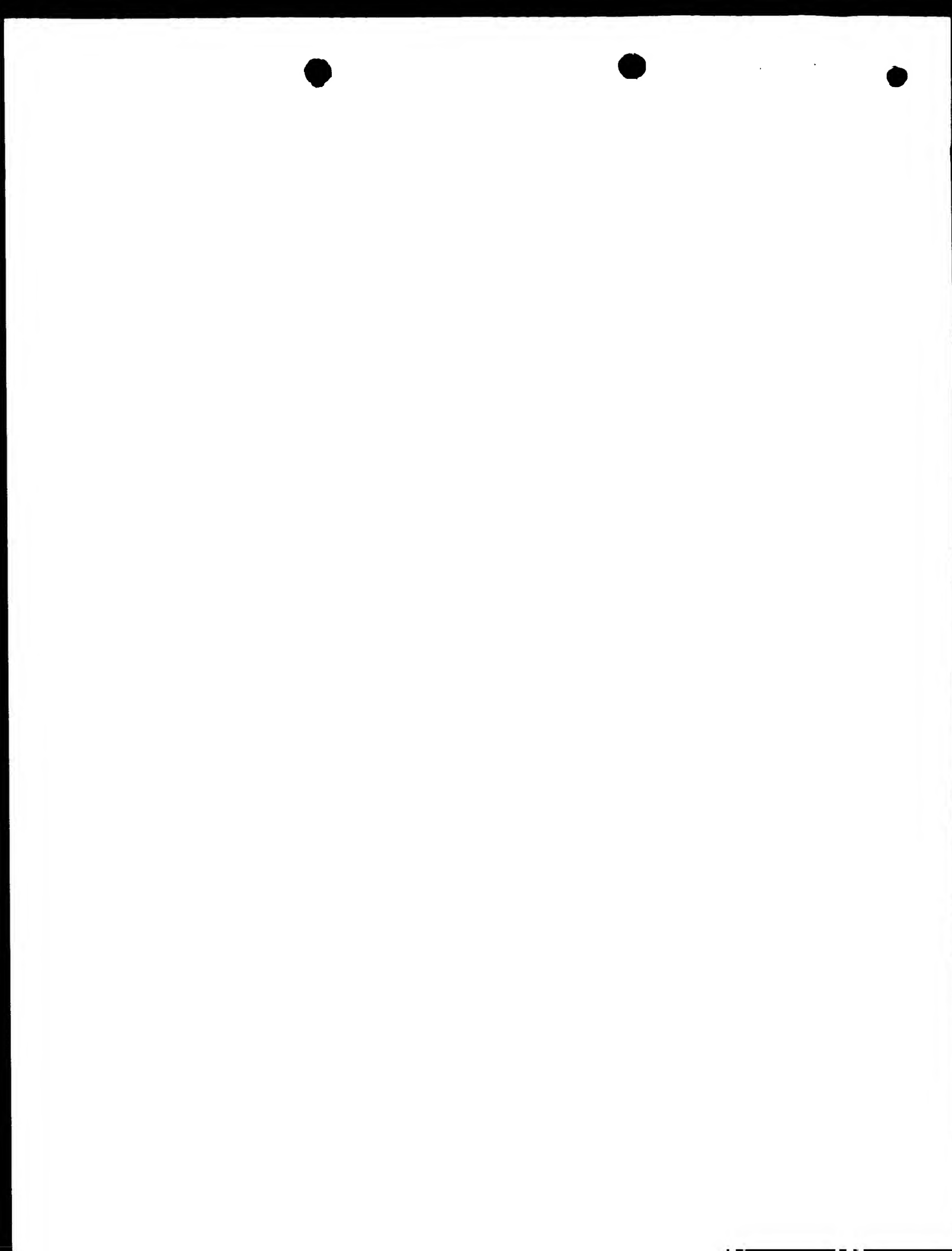
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Signed

Dated 18 April 2000



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19 JUN 1999

21JUN99 E455971-1 D02819  
P01/7700 0.00 - 9914329.9

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Cardiff Road  
Newport  
Gwent NP9 1RH**Request for a patent***(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)*

1. Your reference	C1076/C		
2. Patent application number <i>(The Patent Office will fill in this part)</i>	19 JUN 1999	9914329.9	
3. Full name, address and postcode of the or of each applicant <i>(underline all surnames)</i>	Michael COLE Poplar Farm Low Road Marlesford Woodbridge Suffolk IP13 0AL		
Patents ADP number <i>(if you know it)</i>	642372001		
If the applicant is a corporate body, give the country/state of its incorporation			
4. Title of the invention	Evaporation of Liquids		
5. Name of your agent <i>(if you have one)</i>	Keith W Nash & Co		
"Address for service" in the United Kingdom to which all correspondence should be sent <i>(including the postcode)</i>	90-92 Regent Street Cambridge CB2 1DP		
Patents ADP number <i>(if you know it)</i>	1206001		
6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and <i>(if you know it)</i> the or each application number	Country	Priority application number <i>(if you know it)</i>	Date of filing <i>(day / month / year)</i>
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8. Is a statement of inventorship and of right to grant of a patent required in support of this request? <i>(Answer 'Yes' if:</i> a) <i>any applicant named in part 3 is not an inventor, or</i> b) <i>there is an inventor who is not named as an applicant, or</i> c) <i>any named applicant is a corporate body.</i> <i>See note (d))</i>	No		

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Description 3

Claim(s) -

Abstract -

Drawing(s) 2 + 2 16

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Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (*Patents Form 7/77*)

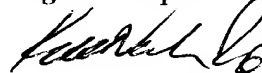
Request for preliminary examination and search (*Patents Form 9/77*)

Request for substantive examination (*Patents Form 10/77*)

Any other documents  
(please specify)

11. I/We request the grant of a patent on the basis of this application.

Signature



Date 18.06.99

Keith W Nash & Co, Agents

12. Name and daytime telephone number of person to contact in the United Kingdom  
Mr Nash (01223) 355477

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C1076.00/C

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The gas may be withdrawn by a suction tube which is preferably maintained close to the surface of the liquid, as the liquid level drops due to evaporation.

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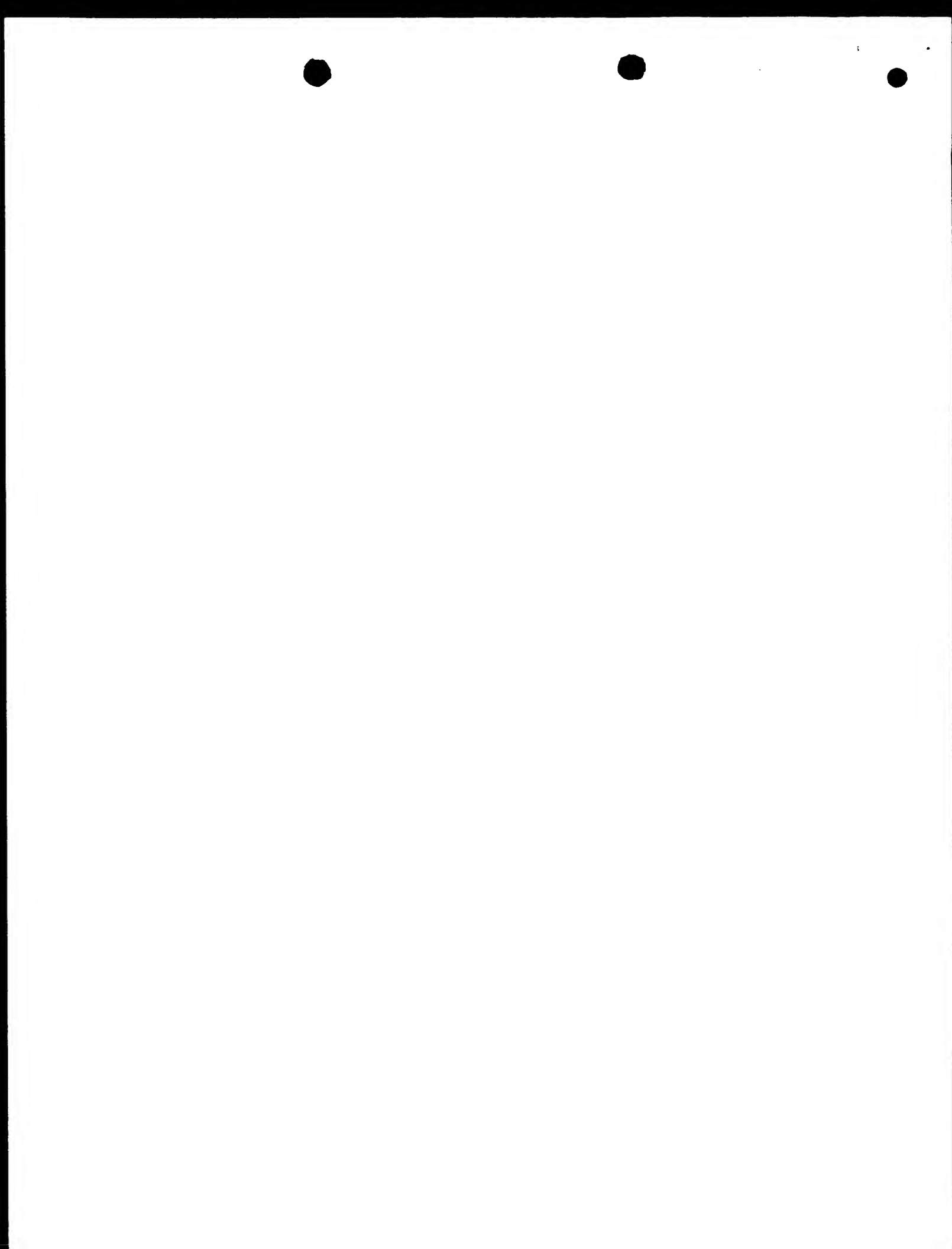
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If the gas from pump 7 is discharged to atmosphere, fresh gas is introduced to the chamber 1 via the pressure control means 15.

The efficiency of the process is enhanced if the tube 5 is progressively lowered as the liquid level drops due to evaporation and held a few millimetres above the sample. This can be achieved manually using a means for lowering the tube 5 or raising the sample 3. Automatic positioning can also be used, using for example optical means to sense the position of the top of the liquid sample and using a sensor system to keep the bottom of the suction tube a few millimetres above the level. Electrical proximity means could also be used to sense the level.

In the example shown in Figure 2 a single sample tube is illustrated. The process can be

applied equally to arrays of many tubes and blocks with numerous wells, for example microtitre plates.



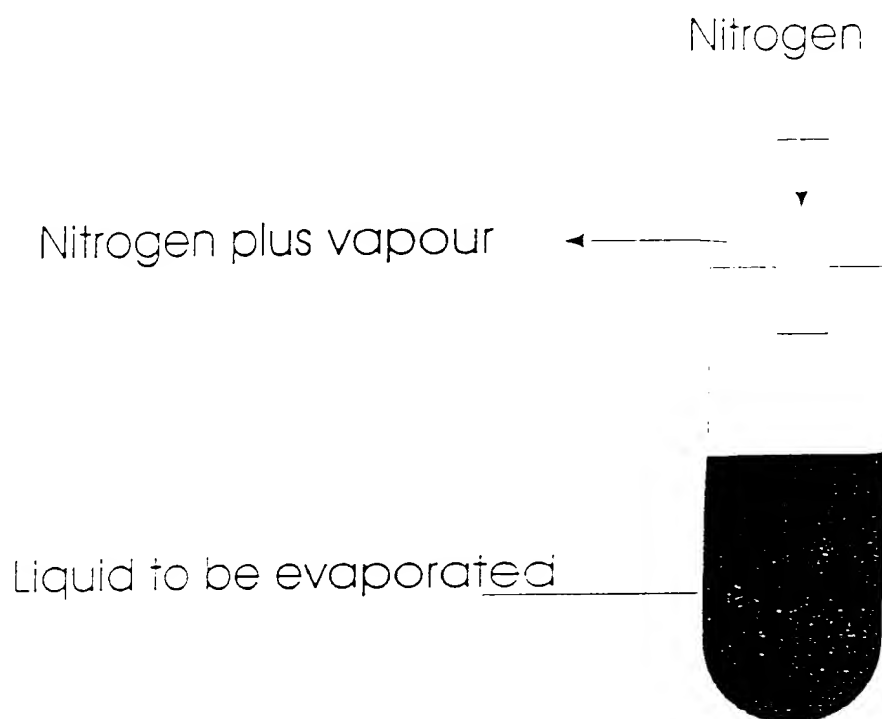
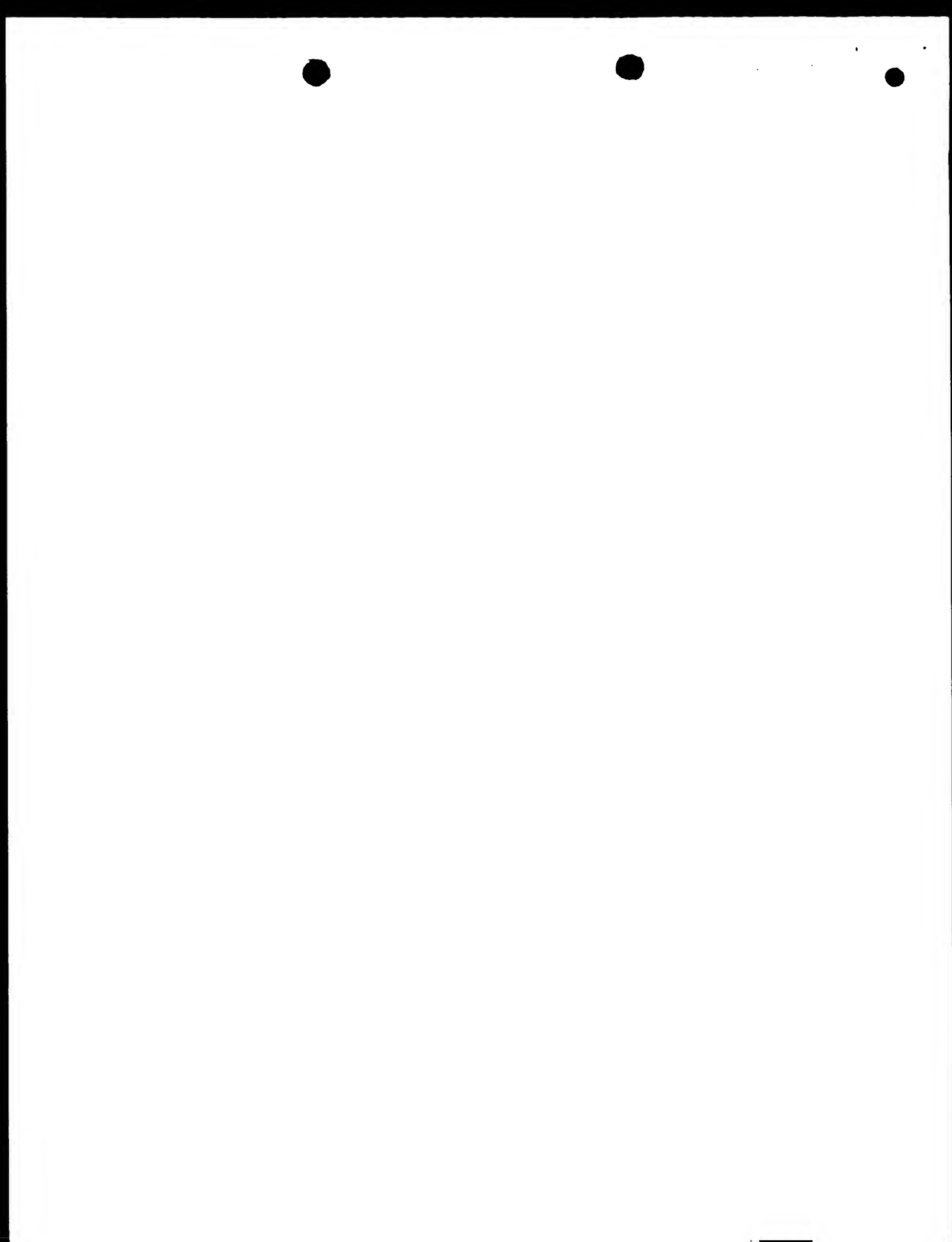


Figure 4- Nitrogen blow-down



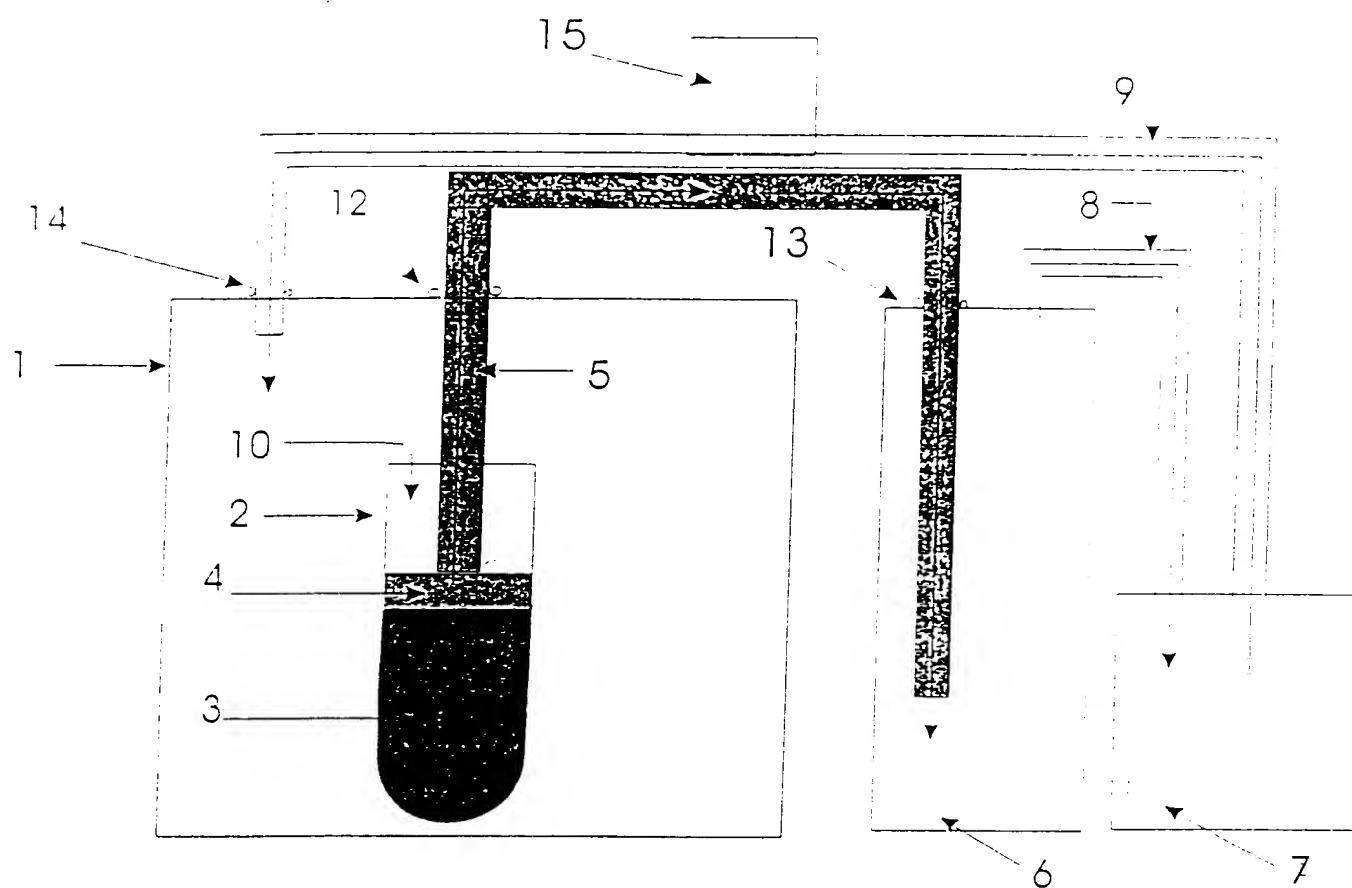
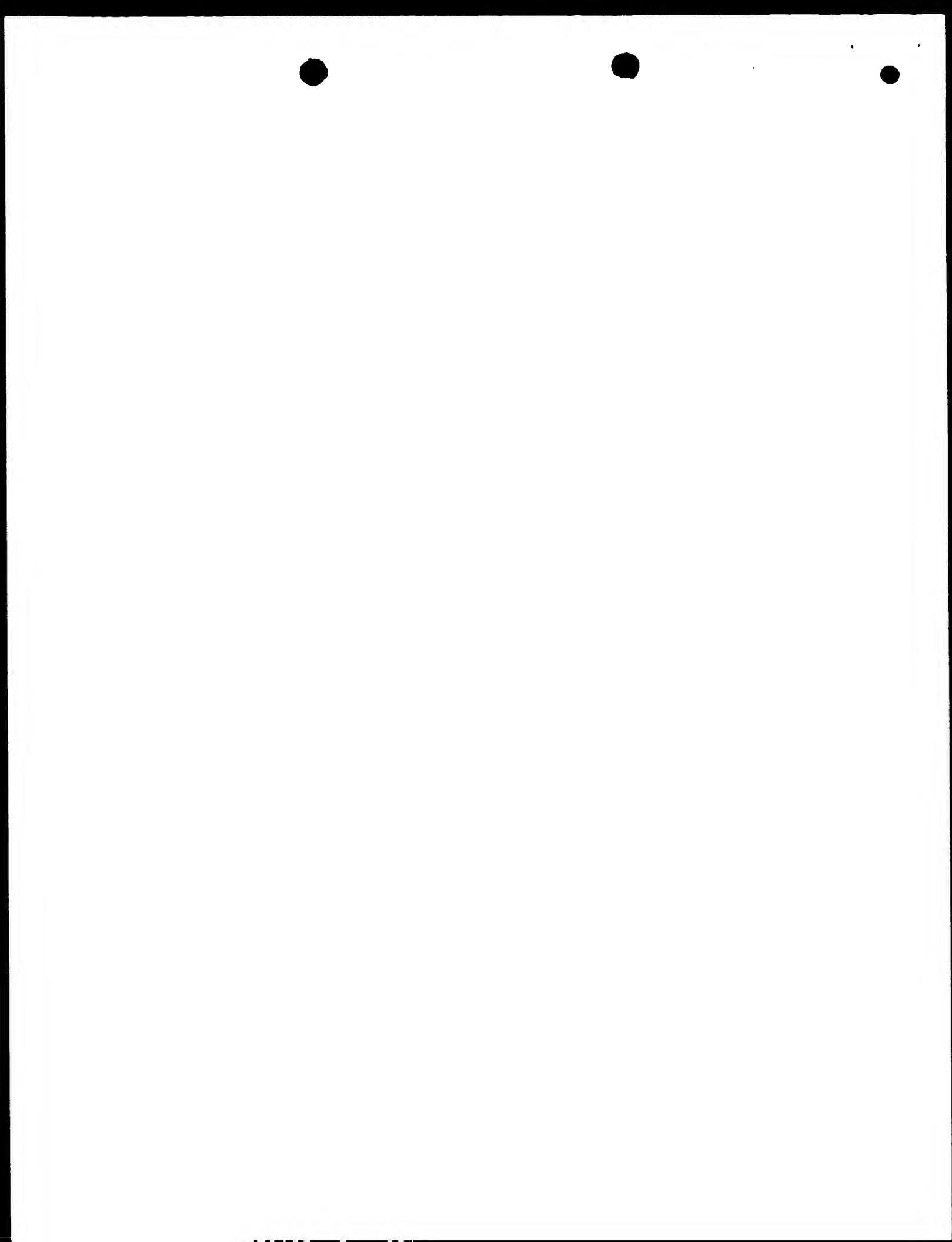
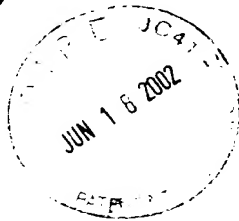


Figure ~~5~~<sub>2</sub> - Evaporation Apparatus







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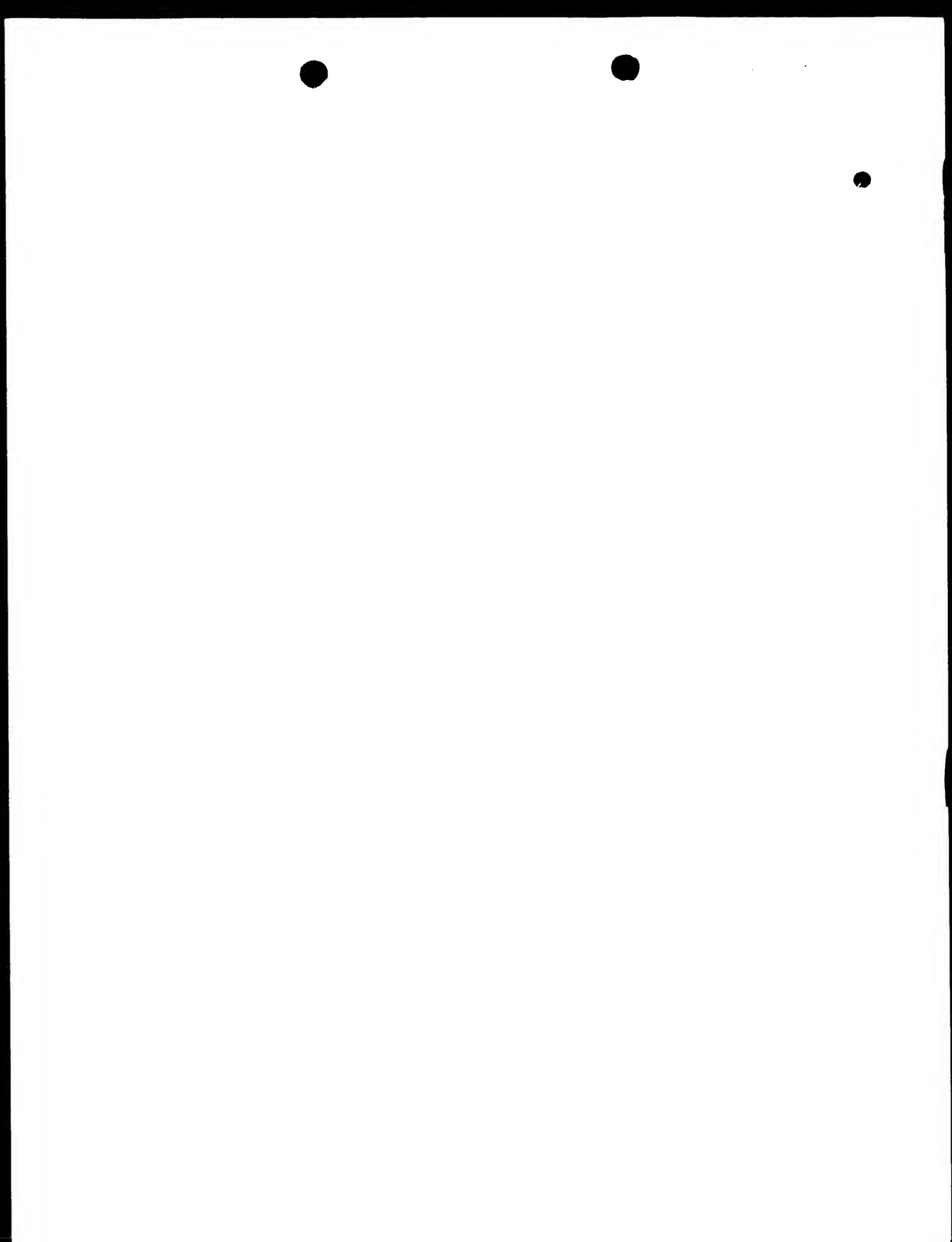
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Dated

13th April 2000



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The Patent Office

Cardiff Road  
Newport  
Gwent NP9 1RH

12 AUG 1999

1. Your reference

KW/DB/0109-00/0

2. Patent application number  
(The Pat

12 AUG 1999

**9918914.4**

3. Full name, address and postcode of the or of each applicant (underline all surnames)

COLE, Michael  
Poplar Farm  
Low Road  
Marlesford  
Woodbridge  
Suffolk  
IP13 0AL

Patents ADP number (if you know it)

If the applicant is a corporate body, give the country/state of its incorporation

642317200

4. Title of the invention

Evaporation of Liquids

5. Name of your agent (if you have one)

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

Keith W Nash & Co  
90-92 Regent Street  
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CB2 1DP

Patents ADP number (if you know it)

1206001

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UK	99087-7.0	17.04.99
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Description	3
Claim(s)	1
Abstract	1
Drawing(s)	2

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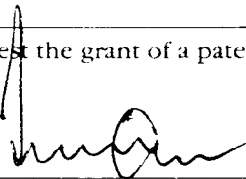
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Request for substantive examination (Patents Form 10/77)

Any other documents (please specify)

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C Dieter Benziger - 01223 355477

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In the example shown in Figure 2 a single sample tube is illustrated. The process can be

applied equally to arrays of many tubes and blocks with numerous wells, for example microtitre plates.

C1076.00/C

Claims

1. A method of evaporating a liquid sample including a mixture of trifluoroacetic acid (TFA) and dichloromethane or another volatile solvent (TFA mix), comprising the steps of continuously removing gas which is rich in vapour from near to top surface of the liquid.
2. A method according to claim 1 in which the gas is cooled in a condenser wherein the vapour condenses, allowing the permanent gas to be recirculated.
3. Apparatus for performing the method of claim 1 or claim 2 in which the liquid is held in a container, and said gas is withdrawn by a suction tube whose open end is maintained close above the top surface of the liquid as the liquid level drops in the container due to evaporation.
4. Apparatus according to claim 3 as dependent upon claim 2 in which the tube is connected to a refrigerated condensing vessel from which the permanent gas is withdrawn by a pump for recirculation.
5. A method of, or apparatus for, evaporating a liquid sample substantially as herein described with reference to, and as shown in, Figure 2 of the accompanying drawings.

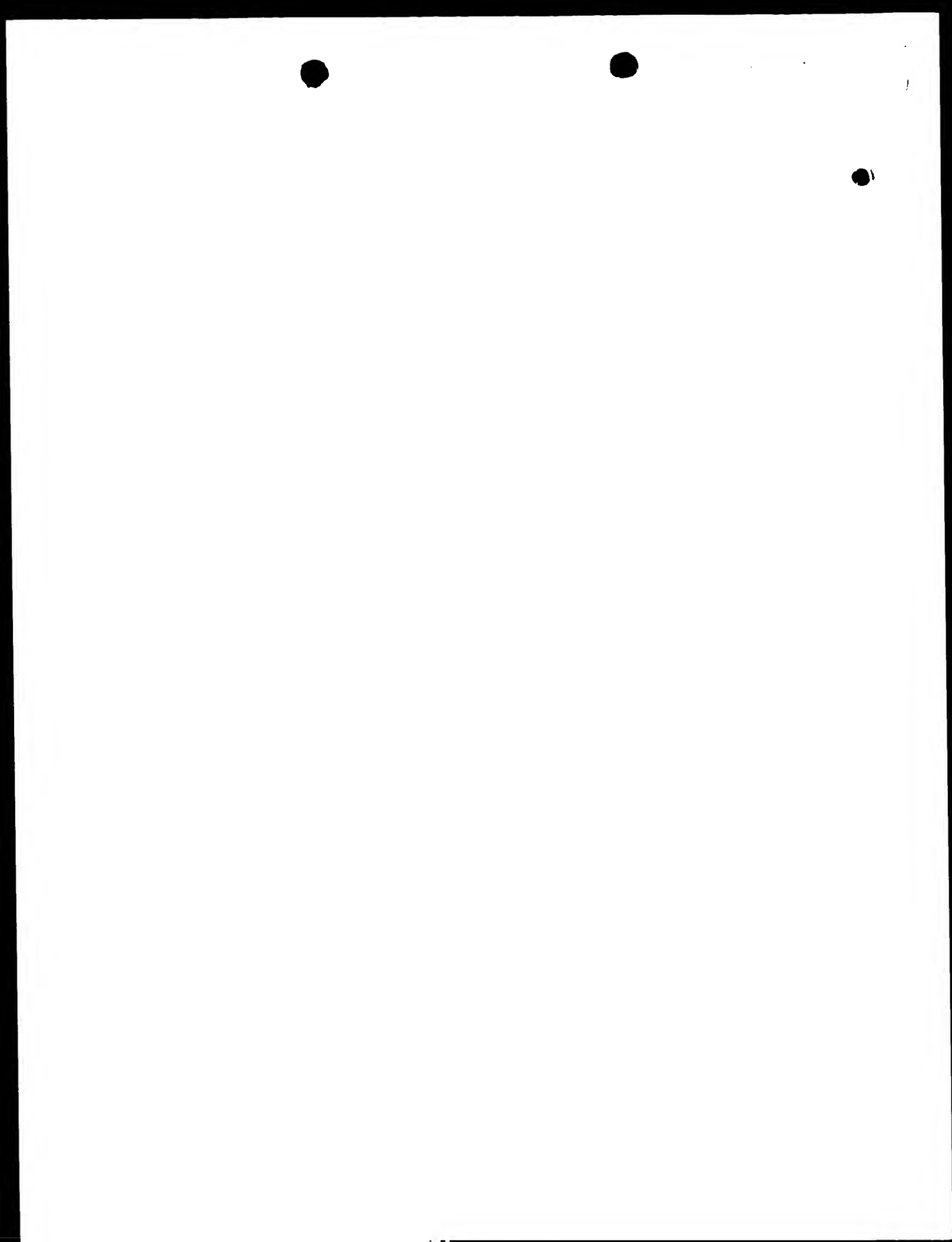


C1076.00/C

Abstract

Title: Evaporation of Liquids

A liquid sample 3 held in an open container 2 and including a mixture of trifluoroacetic acid (TFA) and dichloromethane, or another volatile solvent (TFA mix), is evaporated by continuously removing gas rich in vapour by means of a suction tube 5 whose open end is held close to the top surface of the liquid. Vapour in the gas may then be drawn into a refrigerated condensing vessel 6, from whence the permanent gas may be extracted by a pump 7 for recirculation back into a sealed chamber 1 in which the container is mounted.



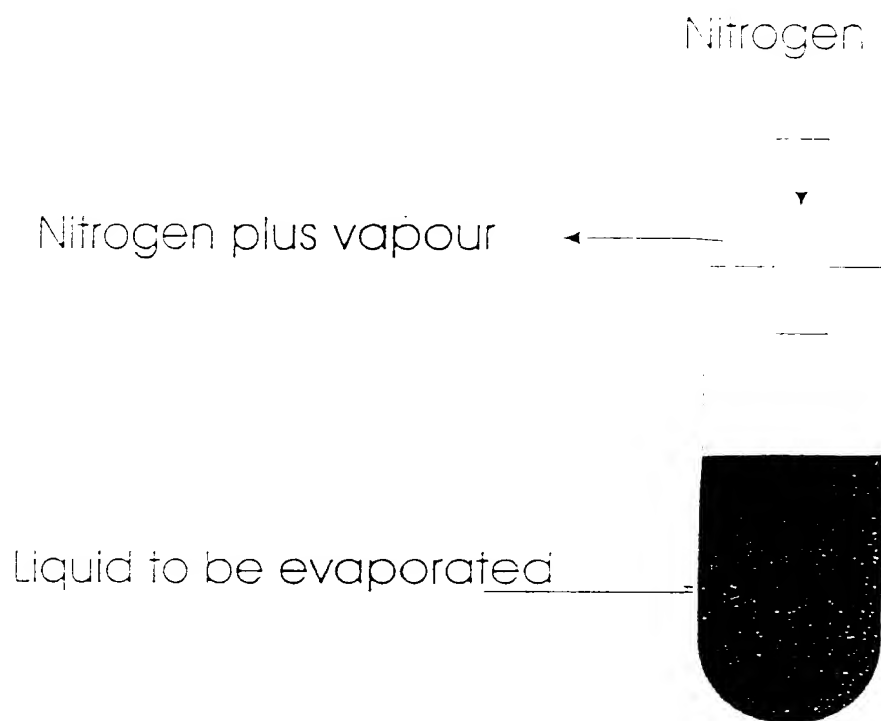


Figure 4- Nitrogen blow-down



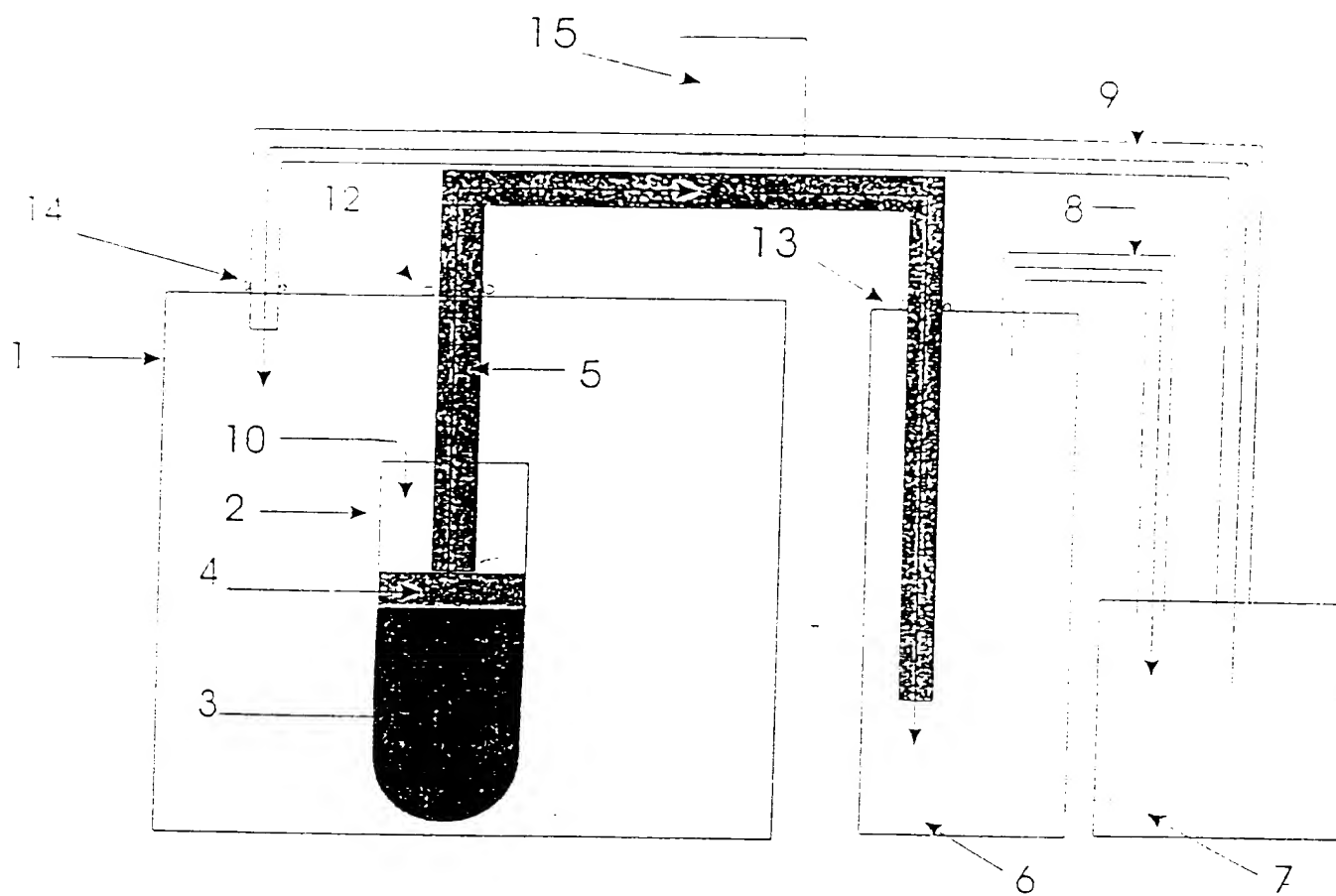
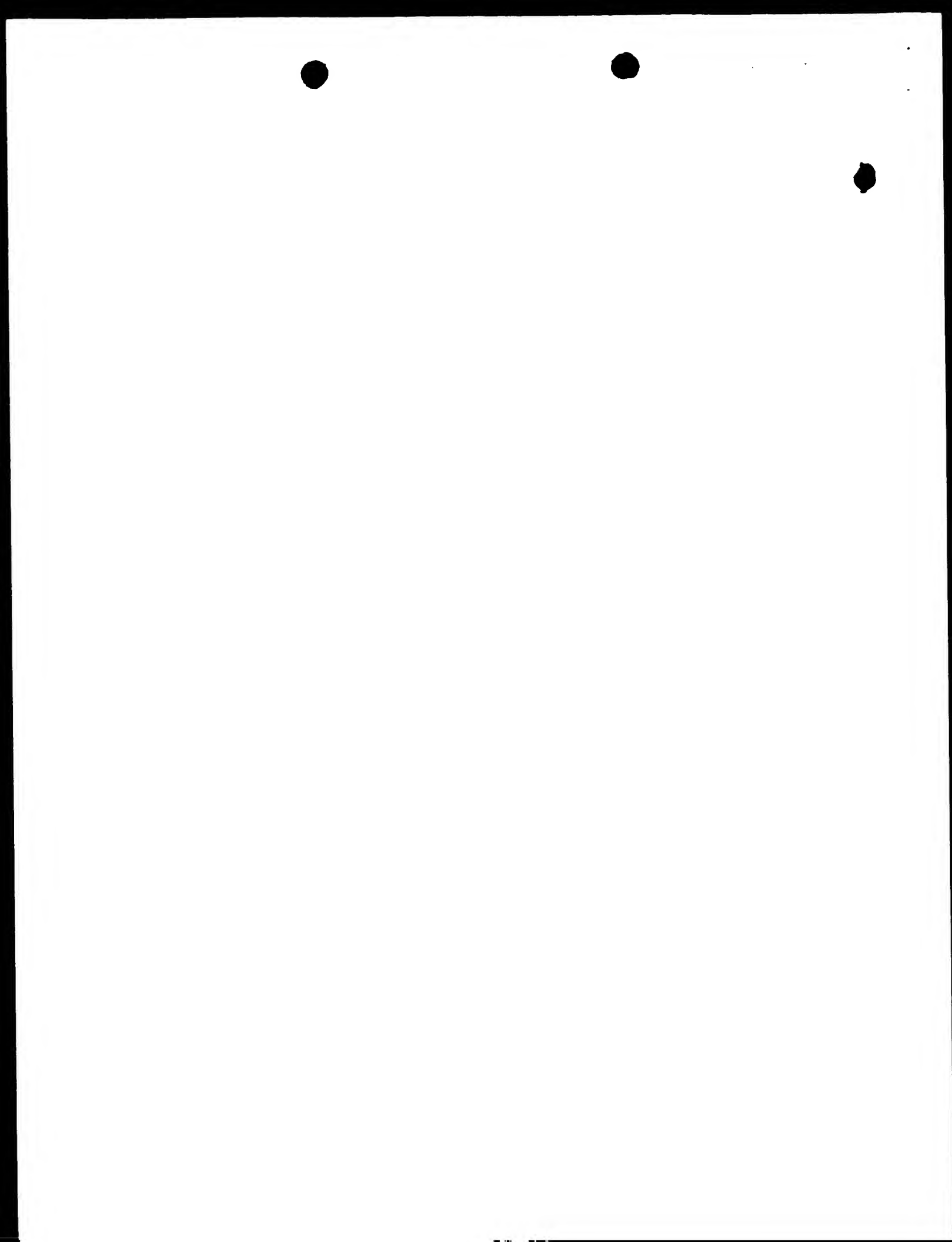
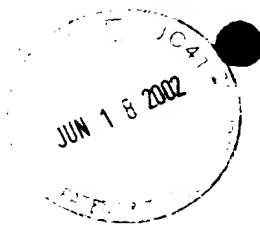


Figure 5 - Evaporation Apparatus





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The Patent Office  
Concept House  
Cardiff Road  
Newport  
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NP10 8QQ

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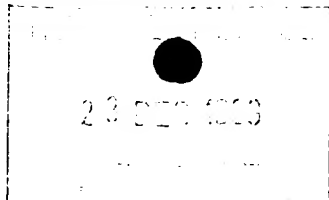
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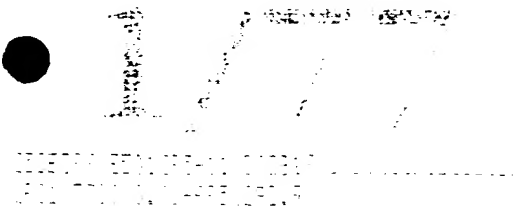
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The Patent Office

Cardiff Road  
Newport  
Gwent NP9 1RH

1. Your reference CI107/C

2. Patent 23 DEC 1999  
(The Patent number) **9930292.9**

3. Full name, address and postcode of the or of each applicant (*underline all surnames*)  
Dr Michael COLE  
Moor Farm  
Friston  
Saxmundham  
IP17 1NH  
United Kingdom  
Patents ADP number (*if you know it*)  
If the applicant is a corporate body, give the country/state of its incorporation

4. Title of the invention  
Evaporation of Liquids

5. Name of your agent (*if you have one*)  
Keith W Nash & Co  
"Address for service" in the United Kingdom to which all correspondence should be sent (*including the postcode*)  
90-92 Regent Street  
Cambridge  
CB2 1DP

Patents ADP number (*if you know it*) 1206001 ✓

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and ( <i>if you know it</i> ) the or each application number	Country	Priority application number ( <i>if you know it</i> )	Date of filing ( <i>day / month / year</i> )
	UK	9908747.0	17.04.99
	UK	9914329.9	19.06.99
	UK	9918914.4	12.08.99

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application  
Number of earlier application  
Date of filing  
(*day / month / year*)

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (*Answer Yes if:*  
a) any applicant named in part 3 is not an inventor, or  
b) there is an inventor who is not named as an applicant, or  
c) any named applicant is a corporate body  
See note (d))  
NO  
A/L  
22-2-2000  
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Patents Form 1/77

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Continuation sheets of this form

Description 4

Claim(s) 2

Abstract 1

Drawing(s) 1

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Priority documents

Translations of priority documents

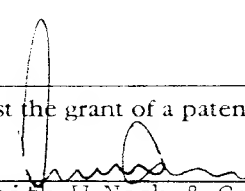
Statement of inventorship and right to grant of a patent (*Patents Form 7/77*)

Request for preliminary examination and search (*Patents Form 9/77*) 1

Request for substantive examination (*Patents Form 10/77*) 1

Any other documents  
(please specify)

11. I/We request the grant of a patent on the basis of this application.

Signature  Date 22.12.99  
Keith W Nash & Co, Agents

12. Name and daytime telephone number of person to contact in the United Kingdom

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C1107.00 C

Title: Evaporation of Liquids

Field of the Invention

This invention concerns evaporation of liquids.

Background to the Invention

Evaporation of a mixture of trifluoroacetic acid (TFA) and dichloromethane, or other volatile solvent, by directing a stream of air or an inert gas such as nitrogen onto the surface of a liquid is a well known process, widely used for evaporating volatile liquids. It suffers from the disadvantage that large volumes of inert gas or air, contaminated with vapour, must be disposed of. This can be expensive if the liquid is inflammable or toxic, as is the case with TFA.

Summary of the Invention

According to the present invention, the inverse of this process may be employed, in which gas, rich in vapour from near the surface of a liquid, is continuously removed.

This allows much easier collection of any toxic or inflammable material.

The gas may be withdrawn by a suction tube which is preferably maintained close to the surface of the liquid, as the liquid level drops due to evaporation.

Advantageously the evaporation rate may be increased by imparting an orbital motion to the container holding the liquid sample, causing the latter to spin around in the form of a vortex.

Preferably the liquid sample is contained within a chamber, and the volume of permanent

gas recirculated to the chamber is progressively reduced, thereby to assist evaporation.

The invention also extends to an apparatus for performing the aforesaid method.

Preferably the container holding the liquid sample is mounted in a vortex evaporator, known per se.

In the drawings, the known process is shown in Figure 1, and an embodiment of the invention is shown in Figure 2.

In Figure 2 a liquid sample 3 in a solvent mixture is contained in a tube 2 inside a sealed chamber 1.

A suction tube 5 is placed with one end 11 in the tube 2 and above the top surface of the sample 3. This tube passes through the chamber 1 through a sealing means 12, through a further sealing means 13, and finally into a sealed refrigerated condensing vessel 6.

A further tube 8 connects the refrigerated condensing vessel 6 to a pump 7 which draws gas (eg air or nitrogen) from the vessel 6 and passes it through a further tube 9 back to the chamber 1 again through a sealing means 14.

A pressure control means 15 allows the pressure in the system to be held at any required level by venting gas to atmosphere or admitting gas into the system from a suitable source, eg a gas cylinder via a pressure regulator.

In operation the pump reduces the pressure in the refrigerated vessel 6, which causes vapour 4 to be drawn into the vessel 6 together with some gas. The temperature of the vessel 6 is maintained at a low enough value to condense the vapour, and any permanent gas passes through the vessel into the pump 7. Gas extracted by the pump 7 could be discharged to the atmosphere because it has been stripped of undesirable vapours in the refrigerated vessel 6 or, as in this illustration, may be recirculated to continue to evaporate the sample.

If the gas from pump 7 is discharged to atmosphere, fresh gas is introduced to the chamber 1 via the pressure control means 15.

Suction by the pump 7 can, and preferably does, result in the pressure within the chamber 1 dropping as the evaporation process continues, in that the volume of air or nitrogen introduced by the control means 15 is arranged to be less than the volume of air or nitrogen withdrawn by the pump 7.

The reduced pressure in the chamber 1 assists in the evaporation of the solvent in the tube 2 in a manner known per se.

The efficiency of the process is enhanced if the tube 5 is progressively lowered as the liquid level drops due to evaporation and held a few millimetres above the sample. This can be achieved manually using a means for lowering the tube 5 or raising the sample 3. Automatic positioning can also be used, using for example optical means to sense the position of the top of the liquid sample and using a sensor system to keep the bottom of the suction tube a few millimetres above the level. Electrical proximity means could also be used to sense the level.

In the example shown in Figure 2 a single sample tube is illustrated. The process can be applied equally to arrays of many tubes and blocks with numerous wells, for example microtitre plates.

In a modification of the apparatus shown in Figure 2, the sample tube 2 is subjected to an orbital motion without alteration of the lateral orientation of the tube, such that the liquid 3 inside the tube moves around the tube in the form of a vortex. Such an arrangement is known per se as a vortex evaporator.

The resultant agitation of the liquid in the tube increases the evaporation rate, allowing a more rapid extraction of vapour-rich gas to take place through the suction tube 5.

A suitable vortex evaporator for this purpose is described in co-pending UK Application

No. 9918914.4 (ref: C1094), the entire disclosure of which is hereby incorporated by reference into the present specification.

C1107.00 C

Claims

1. A method of evaporating a liquid sample including a mixture of trifluoroacetic acid (TFA) and dichloromethane or another volatile solvent, comprising the steps of continuously removing gas which is rich in vapour from near to top surface of the liquid.
2. A method according to claim 1 in which the gas is cooled in a condenser wherein the vapour condenses, allowing the permanent gas to be recirculated.
3. A method according to claim 1 or claim 2 in which the evaporation rate is increased by imparting an orbital motion to the container holding the liquid sample, causing the latter to spin around in the form of a vortex.
4. A method according to claim 2 or claim 3 in which the liquid sample is contained within a chamber, and the volume of permanent gas recirculated to the chamber is progressively reduced, thereby to assist evaporation.
5. Apparatus for performing the method of any one of claims 1 to 3 in which the liquid is held in a container, and said gas is withdrawn by a suction tube whose open end is maintained close above the top surface of the liquid as the liquid level drops in the container due to evaporation.
6. Apparatus according to claim 4 as dependent upon claim 2 in which the tube is connected to a refrigerated condensing vessel from which the permanent gas is withdrawn by a pump for recirculation.
7. Apparatus according to claim 4 or claim 5 in which the container is mounted in a vortex evaporator.

8. Apparatus according to claim 6 or claim 7 further comprising a vacuum chamber in which the container is mounted and means for progressively reducing the volume of permanent gas which is recirculated to the container by the pump.

9. A method of, or apparatus for, evaporating a liquid sample substantially as herein described with reference to, and as shown in, Figure 2 of the accompanying drawings.

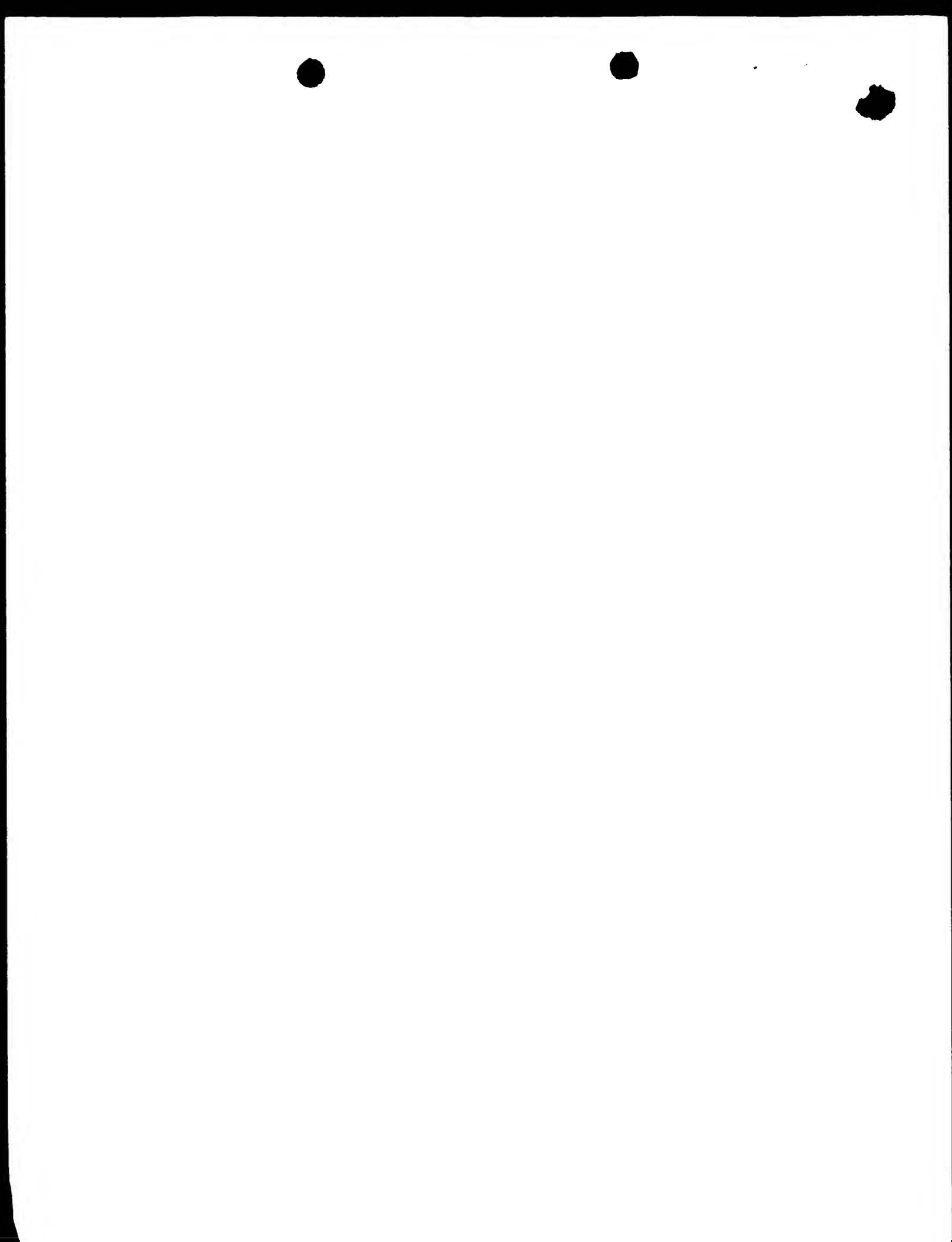


C1107.00/C

Abstract

Title: Evaporation of Liquids

A liquid sample 3 held in an open container 2 and including a mixture of trifluoroacetic acid (TFA) and dichloromethane, or another volatile solvent, is evaporated by continuously removing gas rich in vapour by means of a suction tube 5 whose open end is held close to the top surface of the liquid. Vapour in the gas may then be drawn into a refrigerated condensing vessel 6, from whence the permanent gas may be extracted by a pump 7 for recirculation back into a sealed chamber 1 in which the container is mounted.



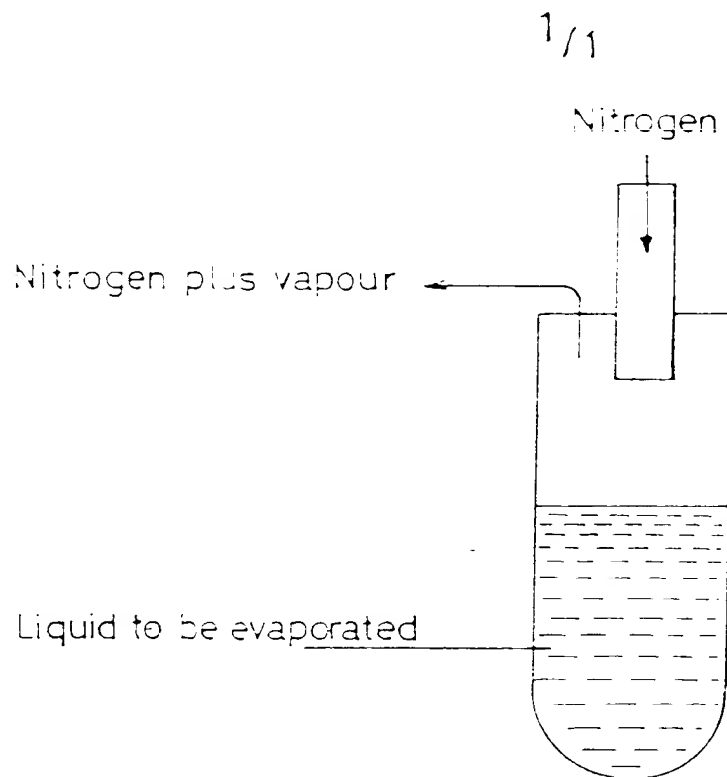


Fig. 1  
Nitrogen blow down

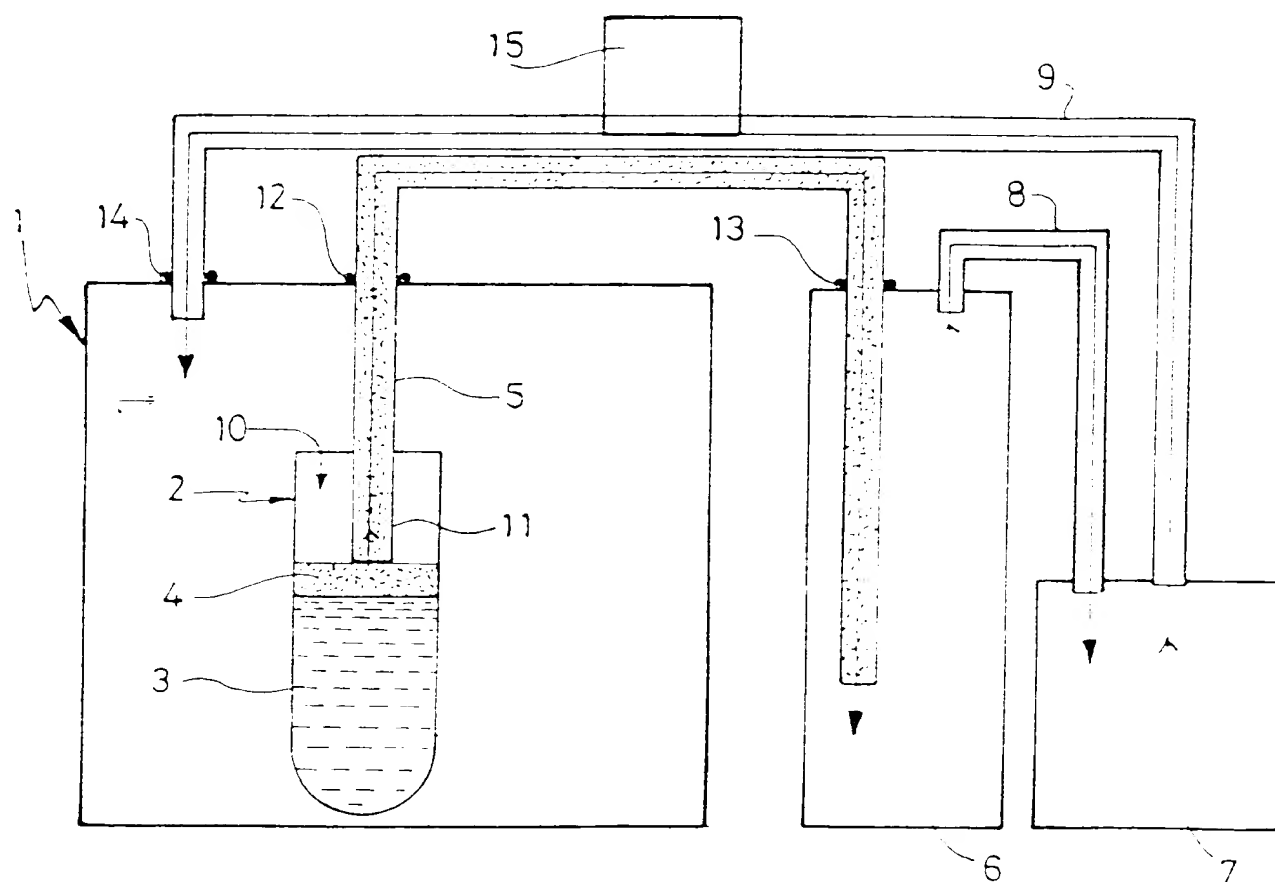


Fig. 2  
Evaporation Apparatus

